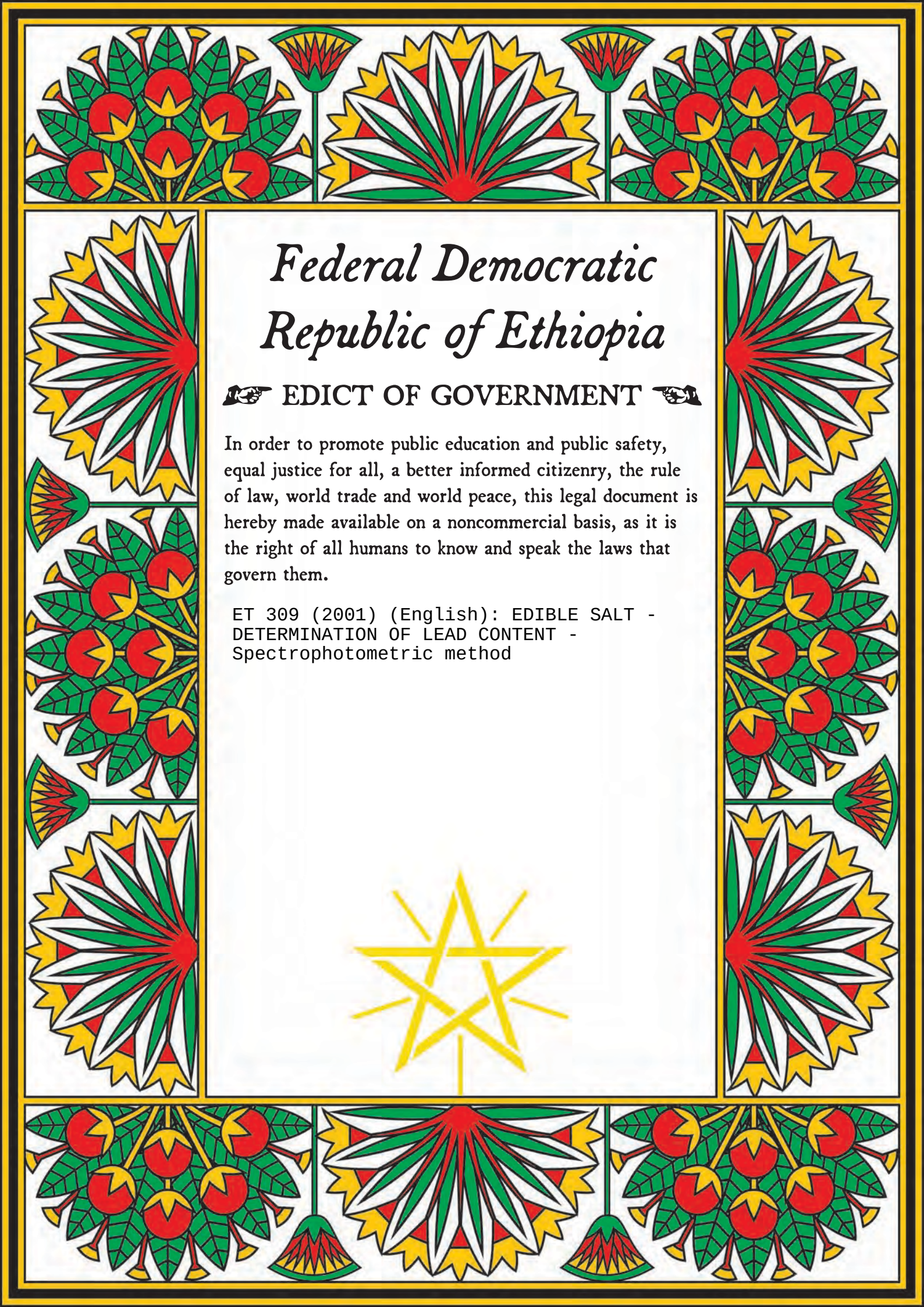




Federal Democratic Republic of Ethiopia

👉 EDICT OF GOVERNMENT 👈

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ET 309 (2001) (English): EDIBLE SALT -
DETERMINATION OF LEAD CONTENT -
Spectrophotometric method



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**Edible salt — Determination of Lead
content — Spectrophotometric method**

ICS: 67.220.20

Published by Ethiopian Standards Agency

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Foreword

This Ethiopian Standard has been prepared under the direction of the Technical Committee for Food additives and Contaminants (TC 21) and published by the Ethiopian Standards Agency (ESA).

The standard is a reaffirmation for reprint of the Ethiopian Standard ES 309:2001, Edible salt - Determination of Lead content - Spectrophotometric method, with some editorial changes without altering the technical contents in the former text.

Edible salt — Determination of lead content — Spectrophotometric method

1 Scope

This Ethiopian Standard specifies the method for the determination of lead content in salt.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute the provisions of this Ethiopian Standard. At the time of publication the edition indicated was valid. All standards are subject to revision and parties to agreements based on this Ethiopian Standard are encouraged to investigate the possibility of applying the most recent edition of the Ethiopian Standard indicated below. Registers of currently valid standards are maintained in the Quality and Standards Authority of Ethiopia.

ES 299:2001, *Edible salt - Sampling*.

3 Principle

The principles are as follows:

- a) The dissolution of the sample in nitric acid solution;
- b) The complexation of certain metals, including lead, by ammonium pyrrolidine-l-carbodithioate, and extraction of the metal complexes into chloroform and back-extraction into nitric acid;
- c) The aspiration of the nitric acid solution into an acetylenepin flame; and
- d) The measurement of the absorbance at a wavelength of approximately 283.3nm, using an atomic absorption spectrophotometer fitted with a lead hollow-cathode lamp.

4 Reagents

The following reagents of recognized analytical reagent quality shall be used:

- a) Nitric acid, ρ 1.40 g/ml, 0.945 g/ml HNO_3 approximately;
- b) Chloroform, ρ 1.48 g/ml;
- c) Ammonium pyrrolidine-l-carbodithioate solution, APCD in 1L of water and filter. Prepare this solution daily;
- d) Ammonia solution, ρ 0.9 g/ml; and
- e) Diammonium hydrogen citrate solution, 113 g/l solution. Dissolve 124g diammonium hydrogen citrate in 500 ml one mark volumetric flask dilute to the mark with water and mix;

Purify this solution as follows:

- Shake 100 ml of the solution with 10 ml of the APCD solution and extract three times respectively with 10 ml, 5 ml and 5 ml of the chloroform.
 - After the phase separation, following each extraction, back extract the combined organic extracts into nitric acid by running the organic layer into a previously dried 100 ml separating funnel containing 1.0 of the nitric acid.
 - Repeat the extraction and back extraction procedure until, when aspirating the nitric acid extracts into the acetylene-air flame, the absorbance measured at approximately 283.3nm is lower than 10 times the absorbance of 20 ml of chloroform extracted with a mixture of 1.0 ml of the nitric acid and 9 ml of water.
 - Transfer the purified solution to a 200 ml one mark volumetric flask, add 20 ml of the ammonia solution, dilute to the mark with water and mix.
- f) Lead stock solution, corresponding to 1000 mg/L Pb. Weigh, to the nearest 0.0001g, 1.598g of lead nitrate $\text{Pb}(\text{NO}_3)_2$, grind it and dissolve it in 10 ml of nitric acid. Transfer the solution quantitatively to 1000 ml one-mark volumetric flask, dilute to the mark with water and mix.

NOTE:

1. 1 ml of this solution contains 0.001 g of lead.
 2. A commercial stock solution for atomic absorption spectrometry of the same strength may also be used if available.
- g) Lead standard solution, corresponding to 10 mg/L Pb. Transfer 10.0 ml of the lead stock solution to a 100 ml one-mark volumetric flask, add 1.0 ml of nitric acid, and then dilute to the mark with water and mix.

5. Apparatus

The following apparatus shall be used:

- a) Ordinary laboratory apparatus;
- b) Separating funnels, 500 ml;
- c) Separating funnels, 100 ml; and
- d) Spectrophotometer.

6. Sampling

Samples shall be taken in accordance with ES 299.

7. Procedure

7.1 General

All the glassware to be employed for this determination shall be washed as follows rinsing very carefully with water after each operation:

- a) with a brush and detergent if the walls are greasy;
- b) with dilute nitric acid.

7.2 Test portion

Weigh, to the nearest 1g, 250g of the test sample.

7.3 Test solution

The test portion is transferred to a 2001 ml beaker, adding 850 ml of water and swirled to dissolve.

7.4 Blank test

850 ml water is also transferred into a 2001 ml beaker.

7.5 Mineralization

To each 2001 ml beaker, 10 ml of nitric acid is added and stirred.

Some glass beads are added, heated to boiling and gently boiled for 30 min.

It is ensured that the total volume never falls below 800 ml, adding more water if necessary. (The same volume to both beakers).

The solution is allowed to cool and quantitatively transferred to a 1000ml one-mark volumetric flask, diluted to the mark with water and mixed.

7.6 Determination

7.6.1 Preparation of the standard matching solutions

- a) Preparation of the standard matching solutions for the test solution

Transfer into each four 500 ml separating funnels, 200 ml of mineralized test solution and the volume of the standard lead solution indicated in table 1.

Complex and extract the lead as described in “b” to “f”

- b) Prepare the standard matching solutions for the blank.

Transfer to four 50ml one-mark volumetric flasks, the volumes of the lead standard solution indicated in table 1. Add to each flask, 5 ml of nitric acid diluted to the mark with water and mix.

- c) Complexation and extraction of lead

Transfer 200 ml of mineralized blank solution to a 500 ml separating funnel.

Table 1 – Mass of lead in standard solutions

Standard matching solution reference number	Volume of the standard lead solution, ml	Corresponding mass of lead, µg
1 ¹⁾	0	0
2	2.5	25
3	5.0	50
4	10.0	100
¹⁾ Test solution		

- d) The procedure is carried out with this 500 ml separating funnel and the four from "a" in the following way.
- 20 ml of the diammonium hydrogen citrate solution
 - 5 ml of the APCD solution is added and shaken for 30 seconds.
 - 10 ml of the chloroform is added and vigorously shaken for 1 min.
- e) After the phase separation the organic layer is run into a previously dried 100 ml separating funnel containing 1 ml of the nitric acid.
- f) This extraction procedure is repeated twice, using 5ml of the chloroform each time and combining the organic phases in separating funnel.
- g) Shake the separating funnel for 30s, add 9ml water and shake for 1min.
- h) When the layers have separated, the lower organic layer is discarded and the upper layer transferred to dry tube.
- i) Apparatus setting

Equip the spectrometer with the lead hollow cathode lamp. Set the lamp current. The slot and the pressure of acetylene and air shall be according to the instrument manufacturer's instructions.

Adjust the wavelength to that of the maximum absorption at approximately 283.3 nm.

j) Spectrometric measurement

Aspirate all the final solutions and the standard matching solutions for the blank into the acetylene - air flame in the following order, and at the following order, and at the same time, each absorbance is determined.

- The four standard matching solutions for the blank test solutions.
- The final blank test solution after the complexation and extraction of lead.
- The three final standard matching solutions after the complexation and extraction of lead.
- The final test solution after C (number 1 from Table 1)

Aspirate water after each measurement.

8. Expression of results

8.1 Calculation

8.1.1 Calibration factors

a) Calibration factor for the test solution

The calibration factors (f_1) of the three standard matching solutions for the test solution (see table 1) is given by the following formula:

$$f_1 = \frac{m_1}{A_1 - A_2}$$

Where,

m_1 is the mass of lead added to the standard matching solution (see number 2, 3 and 4 from table 1 in g)

A_1 is the absorbance obtained for each final standard matching solution

A_2 is the absorbance of the final test solution

b) Calibration factor for the blank test solution

The calibration factor (f_3) of the three standard matching solutions for the blank test is given by the following formula:

$$f_3 = \frac{m}{A_3 - A_4}$$

Where,

m is the mass of lead added to the standard matching solution (see number 2, 3 and 4 from table 1) in g

A_3 is the absorbance obtained for each standard matching solution;

A_4 is the absorbance of the standard matching solution

c) Average calibration factors

The average calibration factors are the arithmetic mean of each group of the three calibration factors found in "b"

d) The lead content, expressed in milligrams of lead (Pb) per kilograms of sodium chloride on moisture free basis, is given by the following formula:

$$\frac{1}{m} \left[\{ (5A_2 \times f_{1m}) - (A_5 \times f_{3m}) \} \left\{ \frac{100}{100 - H} \right\} \right]$$

Where,

A_2 is the absorbance measured for the final test solution;

A_5 is the absorbance measured for the final blank test solution;

m is the mass of the test portion (in g);

f_{1m} is the average calibration factor for the test solution;

f_{3m} is the average calibration factor for the blank test solution.

8.2 Precision

Analysis carried out in three samples lead to the statistical results given in table 2, each laboratory having furnished results obtained by the same operator performing two analysis on each sample.

Table 2 – Statistical results of sodium chloride analysis

Sample	Number of laboratories	Mean, mg Pb/ kg sample	Standard deviation for	
			Repeat ability, σ_r	Reproducibility, σ_R
Rock salt	15	0.045	0.0566	0.1299
Vacuum salt	14	0.025	0.0483	0.1566
Sea salt	15	0.808	0.1254	0.4141

9. Test report

The test report shall include the following information:

- a reference to this Ethiopian Standard;
- the result and method of expression used;
- all information necessary for complete identification of sample;
- any unusual features noted during the determination;
- any operation not included in this Ethiopian Standard or in the Ethiopian Standard to which reference is made, or regarded as optional.



Organization and Objectives

The Ethiopian Standards Agency (ESA) is the national standards body of Ethiopia established in 2010 based on regulation No. 193/2010. ESA is established due to the restructuring of Quality and Standards Authority of Ethiopia (QSAE) which was established in 1970.

ESA's objectives are:-

- ❖ Develop Ethiopian standards and establish a system that enable to check whether goods and services are in compliance with the required standards,
- ❖ Facilitate the country's technology transfer through the use of standards,
- ❖ Develop national standards for local products and services so as to make them competitive in the international market.

Ethiopian Standards

The Ethiopian Standards are developed by national technical committees which are composed of different stakeholders consisting of educational Institutions, research institutes, government organizations, certification, inspection, and testing organizations, regulatory bodies, consumer association etc. The requirements and/or recommendations contained in Ethiopian Standards are consensus based that reflects the interest of the TC representatives and also of comments received from the public and other sources. Ethiopian Standards are approved by the National Standardization Council and are kept under continuous review after publication and updated regularly to take account of latest scientific and technological changes.

Orders for all Ethiopian Standards, International Standard and ASTM standards, including electronic versions, should be addressed to the Documentation and Publication Team at the Head office and Branch (Liaisons) offices. A catalogue of Ethiopian Standards is also available freely and can be accessed in from our website.

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International Involvement

ESA, representing Ethiopia, is a member of the International Organization for Standardization (ISO), and Codex Alimentarius Commission (CODEX). It also maintains close working relations with the international Electro-technical Commission (IEC) and American Society for Testing and Materials (ASTM). It is a founding member of the African Regional Organization for standardization (ARSO).

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